DOCKET NO: 294729USUS0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

•

MAXIME DURAN ET AL.

: EXAMINER:

SERIAL NO: 10/590,197

:

FILED: AUGUST 22, 2006

: GROUP ART UNIT:

FOR: SUBSTRATE SUCH AS A GLASS SUBSTRATE WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPOERTIES

PETITION UNDER 37 C.F.R. § 1.47(a)

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

Arnaud HUIGNARD hereby petitions that the attached Declaration, Power of Attorney and Petition, which is executed by Arnaud HUIGNARD, but not by Maxime DURAN, be accepted by the Patent Office in the above-identified application to satisfy the Declaration requirements of 37 C.F.R. §1.63. The attached Declaration Under 37 C.F.R. § 1.132 establishes that Maxime DURAN refuses to sign the Rule 63 Declaration or cannot be reached. The last known address of Maxime DURAN is:

09/24/2007 GFREY1

00000084 10590197

Maxime DURAN Lotissement Torronbe

Chemin d'Aude

02 FC:1463

200.00 OP

11570 CAVANAC France

The translation between French and English on the Rule 63 Declaration is accurate. The fee set forth in 37 C.F.R. §1.17(g) of \$200.00 is attached. A duplicate copy of this sheet is attached.

Customer Number

Respectfully submitted,

22850

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C.

Taul) lmbach

Tel: (703) 413-3000 Fax: (703) 413-2220

Norman F. Oblon

Fax: (/03) 413-2 (OSMMN 08/03) A

Corwin P. Umbach, Ph.D. Registration No. 40,211

Attachments:

Declaration, Power of Attorney and Petition; Declaration Under 37 C.F.R. §1.132

DOCKET NO: 294729USUS0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

MAXIME DURAN ET AL.

: EXAMINER:

SERIAL NO: 10/590,197

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: GROUP ART UNIT:

FOR: SUBSTRATE SUCH AS A GLASS SUBSTRATE WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPOERTIES

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

WE, Xuan VO and Francois LUCAS, citizens of France, hereby declare and state that:

- 1. We are employed in the patent department of Saint-Gobain Recherche, located at 39 Quai Lucien Lefranc, Aubervilliers, France.
- 2. On May 4, 2007, Xuan VO sent to Maxime DURAN by certified mail the attached letter dated May 3, 2007, signed by Xuan VO and including a copy of the attached Declaration and Power of Attorney for Patent Application. The letter dated May 3, 2007, asked Maxime DURAN to sign and return the same Declaration.
- 3. The attached postal receipt shows that the letter dated May 3, 2007, was delivered to Maxime DURAN.

- 4. On August 5, 2007, Xuan VO mailed to Maxime DURAN by certified mail the attached letter dated July 6, 2007, signed by Francois LUCAS. The letter dated July 6, 2007, included an Assignment and a complete copy of the above-identified application (specification, claims, no drawings, and the attached Declaration). In the letter dated July 6, 2007, the used terminology "declaration of cession" and "pouvoir" refers respectively to the joined "Assignment of Application" and "Power of Attorney". The letter dated July 6, 2007, asked Maxime DURAN to sign and return the Assignment and the Declaration.
- 5. The attached postal receipt shows that the letter dated July 6, 2007, was delivered to Maxime DURAN.
- 6. The letter dated July 6, 2007, mentions the letter dated May 3, 2007, and that Saint-Gobain Recherche had tried several times to reach Maxime DURAN by telephone without success.
- 7. Maxime DURAN has not executed and returned to us the Declaration that was included with the letter dated May 3, 2007.
- 8. Maxime DURAN has not executed and returned to us the Declaration that was included with the letter dated July 6, 2007.
- 9. Maxime DURAN has not responded to the letters dated May 3 and July 6, 2007.
- 10. We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false

statements may jeopardize the validity of this application or any patent issuing thereon.

11. Further declarants saith not

Xuan VO

Date

Date

François LUCAS

Date

Attached:

Letter dated May 3, 2007 (with English translation and translator's certification)

Postal Receipt (with English translation and translator's certification)

Letter dated July 6, 2007

Postal Receipt (with English translation and translator's certification)

Declaration

RWS Group Ltd, of Europa House, Marsham Way, Gerrards Cross, Buckinghamshire, England, hereby declares that, to the best of its knowledge and belief, the following document, prepared by one of its translators competent in the art and conversant with the English and French languages, is a true and correct translation of the accompanying documents in the French language.

Signed this 19th day of September 2007

C. E. SITCH

Managing Director - UK Translation Division

For and on behalf of RWS Group Ltd

SAINT-GOBAIN RECHERCHE

Patents Department

FL/XV-no. 10878-07

Mr Maxime DURAN
Lotissement Torronbe

Chemin d'Aude

11570 CAVANAC

SENT BY POST

(With acknowledgement of receipt)

Aubervilliers, 3 May 2007

Our ref.: FL2 2004022 US

Patent application No. 10/590197 in the name of SAINT-GOBAIN GLASS FRANCE Sending of original power of attorney

Hello Maxime,

To allow us to complete the file for the abovementioned patent application abroad, we would be grateful if you would duly sign the enclosed original powers of attorney and return them to us.

To this end, please find enclosed a stamped envelope.

We trust that this has reached you safely.

Yours sincerely

[signed]

Xuan VO

Patents Department

Tel.: +33-1-48.39.58.98 / Fax +33-1-48.39.66.96

E-Mail: xuan.vo@saint-gobain.com

Enc.: Powers of attorney



Service Brevets FL/XV- n° 10878-07

> M. Maxime DURAN Lotissement Torronbe Chemin d'Aude 11570 CAVANAC

ENVOI PAR COURRIER

(Avec accusé de réception)

Aubervilliers, le 3 mai 2007

Nos réf. : FL2 2004022 US

Demande de brevet d'invention N° 10/590197 au nom de SAINT-GOBAIN GLASS FRANCE Envoi de pouvoir original

Bonjour Maxime,

Afin de compléter le dossier de la demande de brevet à l'étranger citée ci-dessus, nous vous remercions de nous renvoyer les originaux des pouvoirs joints dûment signés.

A cet effet, ci-joint une enveloppe pré-timbrée.

Vous en souhaitant bonne réception, nous vous prions d'agréer l'expression de nos sentiments les meilleurs.

Xuan YÓ

Service Brevets Tel: +33-1-48.39.58.98 / Fax +33-1-48.39.66.96

E-Mail: xuan.vo@saint-gobain.com

PJ: Pouvoirs

Declaration and Power of Attorney for Patent Application Déclaration et Pouvoirs pour Demande de Brevet

French Language Declaration

En tant l'inventeur nommé ci-après, je déclare par le présent acte que:	As a below named inventor, I hereby declare that:
Mon domicile, mon adresse postale et ma nationalité sont ceux figurant ci-dessous à côté de mon nom.	My residence, mailing address and citizenship are as stated next to my name.
Je crois être le premier inventeur original et unique (si un seul nom est mentionné ci-dessous), ou l'un des premiers co-inventeurs originaux (si plusieurs noms sont mentionnés ci-dessous) de l'objet revendiqué, pour lequel une demande de brevet a été déposée concernant l'invention intitulée	I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor(if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.
SUBSTRAT, NOTAMMENT VERRIER, A SURFACE HYDROPHOBE, AVEC UNE DURABILITE AMELIOREE DES PROPRIETES HYDROPHOBES	SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES
et dont la description est fournie ci-joint à moins	the specification of which
☐ ci-joint	is attached hereto.
a été déposée le 23 Février 2005 sous le numéro de demande des États-Unis ou le numéro de demande international 10/590197	was filed on February 23, 2005 as United States Application Number or PCT International Application Number 10/590197
et modifiée le (le cas échéant).	and was amended on (if applicable)
Je déclare par le présent acte avoir passé en revue et compris le contenu de la description ci-dessus, revendications comprises, telles que modifiées par toute modification dont il aura été fait référence ci-dessus.	I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.
Je reconnais devoir divulguer toute information pertinente à	I acknowledge the duty to disclose information which is

material to patentability as defined in Title 37, Code of

Federal Regulations, § 1.56.

la brevetabilité, comme défini dans le Titre 37, § 1.56 du

Code fédéral des réglementations.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119(a)-(d) ou § 365(b) du Code des États-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur ou, en vertu du Titre 35, § 365(a) du même Code, sur toute demande internationale PCT désignant au moins un pays autre que les États-Unis et figurant ci-dessous et, en cochant la case, j'ai aussi indiqué ci-dessous toute demande étrangère de brevet, tout certificat d'inventeur ou toute demande internationale PCT ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

Prior Foreign Application(s)
Demande(s) de brevet antérieure(s) dans un autre pays.

0450343	FR	
(Number)	(Country)	
(Numéro)	(Pays)	
(Number)	(Country)	
(Numéro)	(Pays)	

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 119(e) du Code des États-Unis, de toute demande de brevet provisoire effectuée aux États-Unis et figurant ci- dessous.

(Application No.) (Filing Date) (N° de demande) (Date de dépôt)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des États-Unis, de toute demande de brevet effectuée aux États-Unis, ou en vertu du Titre 35, § 365(c) du même Code, de toute demande internationale PCT désignant les États-Unis et figurant ci-dessous et, dans la mesure où l'objet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande antérieure américaine ou internationale PCT, en vertu des dispositions du premier paragraphe du Titre 35, § 112 du Code des États-Unis, je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la demande antérieure et la date de dépôt de la demande nationale ou internationale PCT de la présente demande:

10/590197	23 Février 2005	
(Application No.)	(Filing Date)	
(N° de demande)	(Date de dépôt)	
(Application No.)	(Filing Date)	
(N° de demande)	(Date de dépôt)	

Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d'une incarcération, ou des deux, en vertu de la § 1001 du Titre 18 du Code des États-Unis, et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby claim foreign priority under Title 35, United States Code, § 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed

	Priority Cla Droit de pr Revendi	riorité	
24/02/04	\boxtimes		
(Day/Month/Year Filed)	Yes	No	
(Jour/Mois/Année de dépôt)	Oui	Non	
(Day/Month/Year Filed)	Yes	No	
(Jour/Mois/Année de dépôt)	Oui	Non	
I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.			

(Application No.) (Filing Date)
(N° de demande) (Date de dépôt)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application,

(Status: Patented, Pending, Abandoned) (Statut : breveté, en cours d'examen, abandonné)

(Status: Patented, Pending, Abandoned)

(Statut : breveté, en cours d'examen, abandonné)

I hereby declare that ail statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

French Language Declaration

POUVOIRS: En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) suivant(s) pour qu'ils poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire s'y rapportant avec l'Office des brevets et des marques: (mentionner le nom et le numéro d'enregistrement).

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact ail business in the Patent and Trademark Office connected therewith: (list name and registration number)



022850

Adresser toute correspondance à:

Send Correspondence to:



022850

Adresser tout appel téléphonique à: (nom et numéro de téléphone)

Direct Telephone calls to: (name and telephone number)

(703)413-3000

Nom complet de l'unique ou premier inventeur DURAN Maxime		Full name of the sole or first inventor DURAN Maxime	
Signature de l'inventeur Date 2 mai 2007		sole or first inventor's signature Date May 2, 20	
Domicile Lotissement Torronbe Chemin d'Aude		Residence Lotissement Torronbe Chemin d'Aude	
Nationalité FRANCE		Citizenship FRANCE	
Adresse Postale 11570 CAVANAC - FRANCE		Mailing Address 11570 CAVANAC - FRANCE	

Nom complet du second co-inventeur, le cas échéant HUIGNARD Arnaud		Full name of second joint inventor, If a HUIGNARD Arnaud	any
Signature de l'inventeur Date 2 mai 2007		Second inventor's signature Date May	
Domicile 33 BIS RUE LAMARCK		Residence 33 BIS RUE LAMARCK	
Nationalité FRANCE		Citizenship FRANCE	
Adresse Postale 75018 PARIS FRANCE		Mailing Address 75018 PARIS FRANCE	

(Fournir les mêmes renseignements et la signature du septième co-inventeur et de tout co-inventeur supplémentaire.)

(Supply similar information and signature for seventh and subsequent joint inventors.)

Assignment of Application

Page 1 of 2 WHEREAS, I (WE) **DURAN Maxime** Lotissement Torronbe INSERT NAMES AND 11570 CAVANAC - FRANCE Chemin d'Aude RESIDENCE **HUIGNARD** Arnaud 33 BIS RUE LAMARCK 75018 PARIS FRANCE ADDRESS OF THE INVENTORS SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A have invented certain new and INSERT TITLE OF useful improvements in: HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF INVENTION: HYDROPHOBIC PROPERTIES for which an application for Letters Patent was executed on INSERT DATE INVENTORS SIGNED (Application No. 10/590197 . filed February 23, 2005), and DECLARATION WHEREAS, SAINT-GOBAIN GLASS FRANCE (hereinafter referred to as "ASSIGNEE") having a place of business at: ADDRESS OF 18 Avenue d'Alsace COMPANY OR 92400 Courbevoie cedex - France OTHER ASSIGNEE

is desirous of acquiring the entire right, title and interest in and to said invention and in and to any Letters Patent that may be granted therefore in the United States and its territorial possessions and in any and all foreign countries;

NOW, THEREFORE, in consideration of the sum of FIVE DOLLARS (\$5.00), the receipt whereof is hereby acknowledged, and for other good and valuable consideration, I (WE), by these presents do sell, assign and transfer unto said ASSIGNEE, the full and exclusive right to the said invention in the United States and its territorial possessions and in all foreign countries and the entire right, title and interest in and to any and all Letters Patent which may be granted therefor in the United States and its territorial possessions and in any and all foreign countries and in and to any and all divisions, reissues, continuations, substitutions and renewals thereof.

I (WE) hereby authorize and request the Patent Office Officials in the United States and its territorial possessions and any and all foreign countries to issue any and all of said Letters Patent, when granted, to said ASSIGNEE as the assignee of my (our) entire right, title and interest in and to the same, for the sole use and behoof of said ASSIGNEE, its (his) successors and assigns, to the full end of the term for which said Letters Patent may be granted, as fully and entirely as the same would have been held by me (us) had this Assignment and sale not been made.

Page 2 of 2

Further, I (WE) agree that I (WE) will communicate to said ASSIGNEE or its (his) representatives any facts known to me (us) respecting said invention, and testify in any legal proceeding, sign all lawful papers, execute all divisional, continuation, substitute, renewal and reissue applications, execute all necessary assignment papers to cause any and all of said Letter Patent to be issued to said ASSIGNEE, make all rightful oaths, and, generally do everything possible to aid said ASSIGNEE, its (his) successors and assigns, to obtain and enforce proper protection for said invention in the United States and its territorial possessions and in any and all foreign countries.

The undersigned hereby grant(s) the firm of Oblon, Spivak, McClelland, Maier & Neustadt, P.C. of 1940 Duke Street Alexandria, Virginia 22314 the power to insert on this assignment any further identification, including the application number and filing date, which may be necessary or desirable in order to comply with the rules of the United States Patent and Trademark Office for recordation of this document.

Date:			
	May 2, 2007	(Signature of Inventor) DURAN Maxime	:
Date:			
	May 2, 2007	(Signature of Inventor) HUIGNARD Arnaud	
Date:			
_		(Signature of Inventor)	
Date: _		<u> </u>	
		(Signature of Inventor)	
Date: _			
		(Signature of Inventor)	
Date: _			
		(Signature of Inventor)	
Date: _		_	

OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C. ATTORNEYS AT LAW 1940 DUKE STREET ALEXANDRIA, VIRGINIA 22314

DEED OF ASSIGNMENT

Korean Patent Application No. 10-2006-7016725

Entitled:

SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES

We/I, DURAN Maxime	· · · · · · · · · · · · · · · · · · ·
of_75020 PARIS	FRANCE
, the undersigned, do hereby	declare that I am the true owner of the invention entitled
as above and that I have assig	gned the entire right and interest to obtain a patent in
respect of said invention unto	SAINT-GOBAIN GLASS FRANCE
of 18 Avenue d'Alsace F- 92400 COURBEVOIE FRANCE	
or their successor so far as K	orea is concerned. Dated this 24th day of August 2006
	DURAN Maxime

申请权转让合同

兹有转让方的专利申请:				
专利申请号:	专利申请日:	年	月	旦
专利申请名称:	,		,	
愿将上述专利申请的权益转让给受让	方 。		, , , , , , , , , , , , , , , , , , , 	THE STATE OF THE S
转让方(原专利申请人)(签章)	受让方(签章)。			
转让方法人代表(签章):	受让方法人代	表(签章):		
第 月 日		3 . (5)		
ASSIGNME DURAN Maxime	NT CONTRAC	<u> </u>		
I/We, the assignor, the owner of	the applicatio	n:	5	
Application Number: PCT/FR2005/050119 Title: SUBSTRATE, SUCH AS A GLASS SUBSTRATE SUCH SUCH AS A GLASS SUBSTRATE SUCH SUCH SUCH SUCH SUCH SUCH SUCH	Filing Date: ATE, WITH A HYDROPHODBIC PROPERTIES Four share to t	23/02//2005 DBIC SURFAC	E AND.	racte
in relation to the above identifie				
of China to the assignee as list				
By Assignor:	By Assignee:			
DURAN Maxime Signature of the Legal	SAINT-GOBA Signature of	the Lega	11.	
Representative of the Assignor:	Representati René MULLER,			B:
Dated this:	Dated this:	24th Augus	2006	

MOMSEN, LEONARDOS & CIA.

Agente da Propriedade Industrial (Licensed Patent and Trademark Agents) RUATEOFILO OTTONI,63- 10° andar – 20090-080 Rio de Janeiro - BRASIL

DOCUMENTO DE CESSÃO Patente

ASSIGNMENT Patent

(1) Insert name(s), full qualification and address of the inventor(s) (of, assignor of priority rights)

DURAN Maxime

Enginneer

Lotissement Torronbe

Chemin d'Aude

11570 CAVANAC - FRANCE

 Inventor of

SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES

Application filed on.. 23/02//2005 under Serial N° PCT/FR2005/050119

(31 insert name(s). full qualifications and address of the physical of juristic person(s) in whose name(s) the case must be filed

para que ainda não obteve (obtiveram) patente tio Governo do Brasil declara(m) peta presente que d á (d ao) seu pleno consentimento para que seja concedida em nome de

SAINT-GOBAIN GLASS FRANCE

18 Avenue d'Alsace F- 92400 COURBEVOIE

FRANCE

a patente correspondente, já requerida ou a requerer no Brasil reivindicando, ou não, a prioridade do(s) pedido(s) acima referido(s) de acordo com a Convenção Internacional. A presente declaração ratifica atos praticados antes do depósito deste psdiüo(s) no Brasil.

for which he (they) has (have) not yet obtained a patent from the Government of Brazil, declare(s) that he (they) give(s) (their) full consent for the Brazilian Patent Office to grant in the name of

SAINT-GOBAIN GLASS FRANCE

18 Avenue d'Alsace F- 92400 COURBEVOIE

FRANCE

the corresponding patent, already applied for or to be applied for, claiming priority, or not of the above mentioned application(s) in accordance with the International Convention. This declaration ratifies acts practised before filing of this (these) case(s) in Brazil.

(4)Insert place and date of execution

(5) Signature(s)

Local Aubervillers,

Data: 24th August

. 3....

2006

Ass.: DURAN Maxime

RWS Group Ltd, of Europa House, Marsham Way, Gerrards Cross, Buckinghamshire, England, hereby declares that, to the best of its knowledge and belief, the following document, prepared by one of its translators competent in the art and conversant with the English and French languages, is a true and correct translation of the accompanying documents in the French language.

Signed this 19th day of September 2007

C. E. SITCH

Managing Director - UK Translation Division

For and on behalf of RWS Group Ltd

Sent from:

Mr MAXIME DURAN Lotissement Torronbe Chemin d'Aude 11570 CAVANAC

RECORDED DELIVERY:

ACKNOWLEDGEMENT OF RECEIPT

LA POSTE

Dispatch number: RA 65 076 722 9 FR

FL2 2004022 US / [illegible]

[stamp]

Return to the following

FRAB

address:

Presented on: / /

Delivered on: 4 / 5 / 07

Signature of the addressee or

of the representative

(Specify surname and first name)

SAINT GOBAIN RECHERCHE

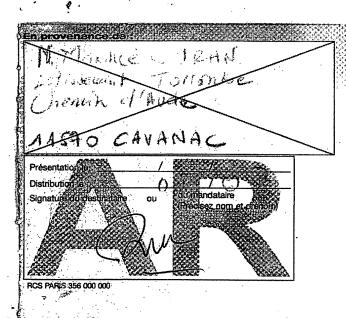
Patents Department

39 Quai Lucien Lefranc

93300 AUBERVILLIERS

[signed]

RCS PARIS 356 000 000

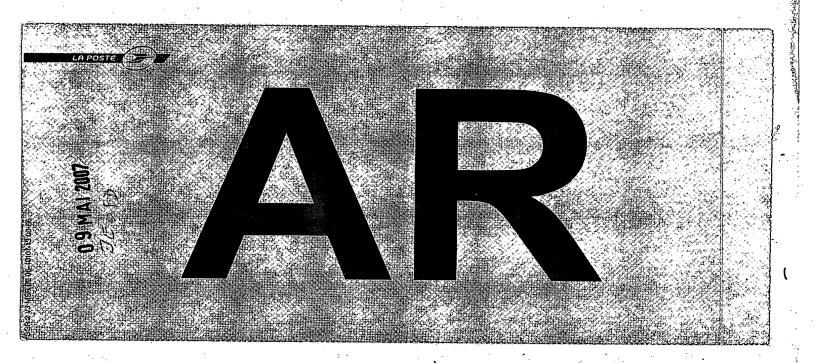


RECOMMANDÉ: **AVIS DE RÉCEPTION**

Numéro de l'envol: RA 65 076 772 9 FR FL2 2004022 US /PV. 40

JAINT GOBAINT RECHERCHE Service Grevets.

39 Quai Lucien Lefranc 93300 AUBERVILLERS





Service Brevets FL/XV- n° 10947-07

M. Maxime DURAN
Lotissement Torronbe
Chemin d'Aude
11570 CAVANAC

SENT BY MAIL (with acknowledgement receipt)

Aubervilliers, July 6, 2007

Nos réf. : FL2 2004022 US

Patent Application N° 10/590197 In the name of SAINT-GOBAIN GLASS FRANCE Declaration of cession

Dear Mr. Maxime DURAN,

Further to our previous certified mail dated May 3, 2007, we did not receive any response from you.

- G. Bourgeois and I tried also to join you by phone several times but without success. Please find again enclosed copy of:
- the specification of the application, as filed in the USA,
- claims as filed in the USA.
- a declaration of cession of the application to Saint-Gobain Glass France
 Please note that we need as soon as possible now your signature on the cession document. The penalty for not complying in due time with the provision of that paper duly signed might be the loss of the right in the United State.

Could you please sign and return the declaration of ¢ession as soon as possible, so that we can provide the USPTO with it?

François LUCAS Saint-Gobain Recherche Saint-Gobain Patent Dept Phone: 33 (0)1 48 39 55 98

Fax: 33 (0)1 48 34 66 96 francois.lucas@saint-gobain.com

PJ: Pouvoirs

Declaration and Power of Attorney for Patent Application Déclaration et Pouvoirs pour Demande de Brevet

French Language Declaration

En tant l'inventeur nommé présent acte que:	ci-après, je déclare par le	As a below named inventor, I hereby declare that:	
Mon domicile, mon adresse ceux figurant ci-dessous à côte	postale et ma nationalité sont é de mon nom.	My residence, mailing address and citizenship are as stated next to my name.	
nom est mentionné ci-desso inventeurs originaux (si plusie	eur original et unique (si un seul us), ou l'un des premiers co- eurs noms sont mentionnés ci- è, pour lequel une demande de ant l'invention intitulée	I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor(if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.	
SUBSTRAT, NOTAMMENT VERRIER, A SURFACE HYDROPHOBE, AVEC UNE DURABILITÉ AMELIORÉE DES PROPRIÉTÉS HYDROPHOBES		SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES	
et dont la description est fourn	e ci-joint à moins	the specification of which	
☐ ci-joint		is attached hereto.	
a été déposée le 23/02 sous le numéro de dema numéro de demande inter PCT/FR2005/050119	ınde des États-Unis ou le	was filed on 23/02//2005 as United States Application Number or PCT International Application Number PCT/FR2005/050119 and was amended on (if applicable)	
Je déclare par le présent acte compris le contenu de la descri comprises, telles que modifiée aura été fait référence ci-dessus	ption ci-dessus, revendications s par toute modification dont il	I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.	
Je reconnais devoir divulguer	toute information pertinente à	I acknowledge the duty to disclose information which is	

material to patentability as defined in Title 37, Code of

Federal Regulations, § 1.56.

la brevetabilité, comme défini dans le Titre 37, § 1.56 du

Code fédéral des réglementations.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119(a)-(d) ou § 365(b) du Code des États-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur ou, en vertu du Titre 35, § 365(a) du même Code, sur toute demande internationale PCT désignant au moins un pays autre que les États-Unis et figurant ci-dessous et, en cochant la case, j'ai aussi indiqué ci-dessous toute demande étrangère de brevet, tout certificat d'inventeur ou toute demande internationale PCT ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

Prior Foreign Application(s)
Demande(s) de brevet antérieure(s) dans un autre pays.

0450343	FR	
(Number) (Numéro)	(Country) (Pays)	
(Number)	(Country)	
(Numéro)	(Pays)	

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 119(e) du Code des États-Unis, de toute demande de brevet provisoire effectuée aux États-Unis et figurant ci- dessous.

(Application No.) (Filing Date) (N° de demande) (Date de dépôt)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des États-Unis, de toute demande de brevet effectuée aux États-Unis, ou en vertu du Titre 35, § 365(c) du même Code, de toute demande internationale PCT désignant les États-Unis et figurant ci-dessous et, dans la mesure où l'objet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande antérieure américaine ou internationale PCT, en vertu des dispositions du premier paragraphe du Titre 35, § 112 du Code des États-Unis, je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la demande antérieure et la date de dépôt de la demande nationale ou internationale PCT de la présente demande:

(Application No.)
(N° de demande)

(Application No.)
(Application No.)
(N° de demande)

(Date de dépôt)

(Date de dépôt)

Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d'une incarcération, ou des deux, en vertu de la § 1001 du Titre 18 du Code des États-Unis, et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby claim foreign priority under Title 35, United States Code, § 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed

•	Priority Claimed	
	Droit de p	riorité
	Revendi	qué
24/02/2004	_ ⊠	
(Day/Month/Year Filed)	Yes	No
(Jour/Mois/Année de dépôt)	Oui	Non
	, □	
(Day/Month/Year Filed)	Yes	No
(Jour/Mois/Année de dépôt)	Oui	Non
I hereby claim the benefit under Title	35, United	States
Code, §119(e) of any United States application(s) listed below.	provisional	٠.

(Application No.) (Filing Date) (N° de demande) (Date de dépôt)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1,56 which became available between the filing date of the prior application and the national or PCT International filing date of this application,

(Status: Patented, Pending, Abandoned) (Statut : breveté, en cours d'examen, abandonné)

(Status: Patented, Pending, Abandoned)

(Statut : breveté, en cours d'examen, abandonné)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

French Language Declaration

POUVOIRS: En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) suivant(s) pour qu'ils poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire s'y rapportant avec l'Office des brevets et des marques: (mentionner le nom et le numéro d'enregistrement).

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact ail business in the Patent and Trademark Office connected therewith: (list name and registration number)



022850

Adresser toute correspondance à:

Send Correspondence to:



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Nom complet de l'unique ou premier inventeur HUIGNARD Arnaud				Full name of the sole or first inventor HUIGNARD Arnaud		
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Signature de l'inventeur	Date		second inventor's signature	Date	
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(Fournir les mêmes renseignements et la signature du septième co-inventeur et de tout co-inventeur supplémentaire.)

(Supply similar information and signature for seventh and subsequent joint inventors.)

Assignment of Application

Page 1 of 2

	WHEREAS, I (WE)	8					
INSERT NAMES AND RESIDENCE	HUIGNARD Arnaud	33 bis Rue Lam	arck		75018 PARIS		FRANCE
ADDRESS OF THE INVENTORS:	DURAN Maxime	LOTISSEMENT	LA TORONI	DE CHEMIN D'AUDE	11570 CAVANAC		FRANCE
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INSERT TITLE OF	have invented certain ne	w and useful improven	nents in:			,	· · ·
INVENTION:	SUBSTRATE, SUCH AS A GLA	SS SUBSTRATE, WITH A H	YDROPHOBIC	SURFACE AND IMPROV	ED DURABILITY OF HYDI	ROPHOBIC	PROPERTIES
INSERT DATE	for which an application	for Letters Patent was	executed on	l			
INVENTORS SIGNED DECLARATION	(Application No. PCT/	FR2005/050119	, filed	23/02//2005), a	nd
INSERT NAME AND	WHEREAS, SAINT-GO						
ADDRESS OF	(hereinafter referred to as	s "ASSIGNEE") havin	g a place of	business at:			
COMPANY OR	18 Avenue d'Alsace						
OTHER ASSIGNEE	F- 92400 COURBEVOIL is desirous of acquiring t	= he entire right, title an	d interest in	and to said invention	on and in and to any	Letters I	Patent that

may be granted therefore in the United States and its territorial possessions and in any and all foreign countries;

NOW, THEREFORE, in consideration of the sum of FIVE DOLLARS (\$5.00), the receipt whereof is hereby acknowledged, and for other good and valuable consideration, I (WE), by these presents do sell, assign and transfer unto said ASSIGNEE, the full and exclusive right to the said invention in the United States and its territorial possessions and in all foreign countries and the entire right, title and interest in and to any and all Letters Patent which may be granted therefor in the United States and its territorial possessions and in any and all foreign countries and in and to any and all divisions, reissues, continuations, substitutions and renewals thereof.

I (WE) hereby authorize and request the Patent Office Officials in the United States and its territorial possessions and any and all foreign countries to issue any and all of said Letters Patent, when granted, to said ASSIGNEE as the assignee of my (our) entire right, title and interest in and to the same, for the sole use and behoof of said ASSIGNEE, its (his) successors and assigns, to the full end of the term for which said Letters Patent may be granted, as fully and entirely as the same would have been held by me (us) had this Assignment and sale not been made.

Further, I (WE) agree that I (WE) will communicate to said ASSIGNEE or its (his) representatives any facts known to me (us) respecting said invention, and testify in any legal proceeding, sign all lawful papers, execute all divisional, continuation, substitute, renewal and reissue applications, execute all necessary assignment papers to cause any and all of said Letter Patent to be issued to said ASSIGNEE, make all rightful oaths, and, generally do everything possible to aid said ASSIGNEE, its (his) successors and assigns, to obtain and enforce proper protection for said invention in the United States and its territorial possessions and in any and all foreign countries.

The undersigned hereby grant(s) the firm of Oblon, Spivak, McClelland, Maier & Neustadt, P.C. of 1940 Duke Street Alexandria, Virginia 22314 the power to insert on this assignment any further identification, including the application number and filing date, which may be necessary or desirable in order to comply with the rules of the United States Patent and Trademark Office for recordation of this document.

Date:	August 1rst 2007				
			HUIGNARD Arnaud	1	
		•			
Date:	August 1rst 2007				
		(Signature of Inventor)	DURAN Maxime		-
Date:					
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SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES

- 5 The present invention relates to a substrate, especially a glass substrate, the surface of which has been rendered hydrophobic, with improved durability of the hydrophobic properties.
- 10 Hydrophobic properties are sought for windows and windshields in the transport field, in particular for motor vehicles and aircraft, and also for glazing in the building industry.
- For applications in the transport field, rain-repellent properties are sought, the water droplets on windshields thus having to easily roll off the glass wall so as to be removed, for example when the vehicle is in motion due to the effect of the air or wind, and to do so with the purpose of improving visibility and, consequently safety, or for facilitating cleaning, or for easily defrosting, etc.
- For applications in the building field, the aim is essentially to make cleaning easier.

For this purpose, the aim is to have an angle of contact of a water droplet with the substrate that is greater than 60° or 70°, the water droplet having not to be flattened or spread out. This is because glazing is said to be functional as long as this angle is greater than 60° in the case of aircraft, and greater than 70° in the case of automobiles. However, in practice this angle should in all cases exceed 90°, the ideal being to obtain droplets that roll off, allowing the water to be removed so quickly as to be able to dispense as far as possible with windshield wipers in the automotive field.

Moreover, the improvement in hydrophobic properties sought must not be to the detriment of the preservation of the other properties, such 5 resistance to mechanical stresses: resistance to shear friction (standardized Opel test, carried out abrasion resistance (Taber test), resistance to wiping by wipers (test simulating the cycles of wiper action); resistance to environmental stresses (WOM test of UVA 10 resistance, or Xenon test; QUV test of UVB resistance for aircraft; NSS (neutral salt spray) resistance test; resistance to chemical stresses: test of resistance to acid and basic detergents; and the optical properties.

15 To render a glass hydrophobic it is known to coat it with a dense silica mineral layer serving as primer for grafting of molecules having hydrophobic properties, such as fluorosilane molecules. European patent EP 0 545 201 describes the application of a dense SiO_2 layer applied by magnetron sputtering, 20 layer being subsequently coated SiO_2 hydrophobic agent.

The filing company has discovered that the hydrophobic properties of such a structure can be further improved, in particular in their durability, with the other properties mentioned above being at least maintained, or even sometimes improved, if the coating of molecules having hydrophobic properties is applied while this layer is in an activated surface state, this activation being able to be produced either by the actual conditions under which the mineral layer is deposited, or by a specific activation treatment.

Thus, the mineral layer (which is the sublayer in the resulting final structure) may be deposited by vacuum sputtering, especially magnetron sputtering, under conditions that allow the layer to be left in an

unstable surface state, with the hydrophobic coating being applied while the surface is still in this state (generally applied immediately), or by a specific activation treatment (plasma excitation, etc.).

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A first subject of the present invention is therefore a substrate, at least one part of the surface of which has been rendered hydrophobic, having for this purpose a hydrophobic surface structure comprising an essentially mineral silicon-containing sublayer and an outer layer of hydrophobic agent grafted onto said sublayer, characterized in that said sublayer has received the outer layer of hydrophobic agent although it had a surface that was in an activated state before being brought into contact with said hydrophobic agent.

The term "activated" is understood to mean that said surface has undergone a treatment which has modified its electrostatic state (by production of charges) and/or its chemical state (creation or destruction of 20 chemical functional groups), in order to increase the reactivity of said surface, which treatment may go as far as tearing the material of the surface, creating irregularities. Moreover, as will be indicated 25 below, the layer of silicon-containing mineral material will constitute the sublayer in the structure may be obtained under conditions in which it is directly in the activated state.

30 The sublayer may be a hard sublayer.

The substrate is especially formed by, or comprises in its part intended to bear said mineral sublayer, a plate, whether plane or with curved faces, of monolithic or laminated glass, of glass-ceramic or of a hard thermoplastic, such as polycarbonate. The glass may be a toughened glass. An example of a curved plate is a windshield. This may be in the assembled state.

The sublayer of the hydrophobic coating may form part of the substrate, the latter being formed by a plate, whether plane or with curved faces, of monolithic or laminated glass or of glass-ceramic, the composition of which, at least on the surface, corresponds to that of the essentially mineral silicon-containing sublayer. An example of a substrate having such an integrated sublayer is a glass dealkylized at least on its surface. International applications WO-94/07806 and WO-94/07807 describe this technology.

The silicon-containing sublayer is especially formed by a compound chosen from SiO_x , where $x \le 2$, SiOC, SiON, 15 SiOCN and Si_3N_4 , it being possible for hydrogen to be combined in all proportions with SiO_x , where $x \le 2$, SiOC, SiON and SiOCN. It may also contain aluminum, in particular up to 8% by weight, or carbon, Ti, Zr, Zn and B.

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Mention may also be made of sublayers consisting of scratch-resistant lacquers, such as polysiloxanes, which have been applied as coating on polycarbonate substrates.

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The silicon-containing sublayer when its surface is in the activated state has a thickness of between 20 nm and 250 nm, especially between 30 nm and 100 nm and in particular between 30 nm and 75 nm. It may have an RMS roughness of between 0.1 nm and 40 nm, in particular between a few nm and 30 nm. It may have an actual developed area at least 40% greater than the initial plane area. Under an SEM microscope, said sublayer may have the appearance of pumistone or of islands.

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Moreover, the silicon-containing sublayer when its surface is in the activated state advantageously has a hardness such that it does not delaminate after 100

revolutions, and preferably up to 200 revolutions, in the Taber test.

The hydrophobic agent may be chosen from:

5 (a) alkylsilanes of formula (I):

 $CH_3(CH_2)_nSiR_mX_{3-m}$

in which:

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- n ranges from 0 to 30, more particularly from 0 to 18;

(I)

- m = 0, 1, 2 or 3;
 - R represents an optionally functionalized organic chain; and
 - X represents a hydrolyzable residue, such as an OR^0 residue, where R^0 represents hydrogen; or a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue;
 - (b) compounds with grafted silicone chains, such as for example $(CH_3)_3SiO[Si(CH_3)_2O]_q$, with no specific limitation as regards the chain length (value of q) and the method of grafting;
 - (c) fluorosilanes, such as those of formula (II): $R^{1}-A-SiR_{p}^{2}X_{3-p}$ (II)

in which:

- - A represents a hydrocarbon chain, optionally interrupted by a heteroatom such as O or S;
 - R^2 represents a linear, branched or cyclic, especially C_1-C_8 , alkyl residue, or an aryl residue;
- X represents a hydrolyzable residue, such as an OR^3 residue, where R^3 represents hydrogen or a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue;

and - p = 0, 1 or 2.

An example of an alkylsilane of formula (I) is octadecyltrichlorosilane (OTS).

The preferred hydrophobic agents are fluorosilanes (c), in particular those of formula (II), particular examples of the latter being those of formula:

10 $CF_3 - (CF_2)_n - (CH_2)_2 - Si(R^4)_3$

in which:

- R4 represents a lower alkyl residue; and
- n is between 7 and 11.
- 15 The layer of hydrophobic agent has for example a thickness of between 1 and 100 nm, preferably between 2 and 50 nm.
- The layer of fluorosilane may have a weight per unit area of grafted fluorine of between $0.1~\mu g/cm^2$ and $3.5~\mu g/cm^2$, in particular between $0.2~\mu g/cm^2$ and $3~\mu g/cm^2$.
- The subject of the present invention is also a process for manufacturing a substrate as defined above, characterized in that a coating layer of hydrophobic agent is deposited, in at least one pass, on the surface of a silicon-containing mineral layer formed at least partly on the surface of the substrate, said deposition of the hydrophobic agent taking place while said surface is in the activated state.

An activated surface of the silicon-containing mineral layer may be obtained by depositing it under conditions in which its surface is obtained directly in the activated state. This is what occurs if a silicon-containing layer is deposited, cold, by PECVD (plasma enhanced chemical vapor deposition) or by magnetron

and/or ion-beam sputtering.

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This is because, in such processes, the growth of the layer takes place using reactive species (ions, radicals, neutrals, etc.) which combine to form the coating. The surface of the coating is therefore by nature in an off-equilibrium state. In addition, this layer may be directly in contact with the plasma gas during growth, which will further increase the activity of the surface and its reactivity (as in the PECVD process).

It is also possible to obtain an activated surface of the silicon-containing mineral layer by carrying out an activation treatment in at least one pass.

Advantageously, the hydrophobic agent is deposited within the shortest possible time, preferably between 1 second and 15 minutes, after the activated surface has been obtained.

activation treatment may be carried out under conditions that do not go as far as etching, by the use plasma or an ionized gas, at reduced 25 atmospheric pressure, chosen from air, oxygen, nitrogen, argon, hydrogen, ammonia and mixtures thereof, or by the use of an ion beam.

Ιt is possible also to carry out an activation 30 treatment under conditions that allow a siliconcontaining layer to be etched, by the use of a plasma of at least one fluorine-containing gas chosen from SF_6 , CF_4 , C_2F_6 and other fluorinated gases, where appropriate combined with oxygen, it being possible for the oxygen to represent up to 50% by volume of the etching plasma. 35

Moreover, according to the present invention, the activation carried out under conditions that allow the

silicon-containing layer to be etched by an activation treatment, which does not cause additional etching but does still modify the chemical nature and/or the electrostatic state of said layer, may be monitored.

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The silicon-containing layer may be deposited, cold, on the substrate by vacuum cathode sputtering, preferably magnetron sputtering and/or ion beam sputtering, or by low-pressure or atmospheric-pressure PECVD, or else deposited hot by pyrolysis.

As examples of the deposition of the SiO₂ sublayer, the following method of implementation may be mentioned, in which: a layer of SiO_2 is deposited on bare glass or on 15 an assembled windshield by PECVD, using a mixture of an nonorganic, silicon-containing precursor, such as SiH4, hexamethyldisiloxane (HMDSO), tetraethoxysilane (TEOS) and 1,1,3,3tetramethyldisiloxane (TMDSO), and an oxidizer $(O_2, NO_2,$ 20 ${\rm CO}_2)$, the subsequent activation being carried out in the same chamber or in a separate chamber.

The hydrophobic agent layer may be deposited by wipingon, evaporation or spraying of a solution containing the hydrophobic agent, or by dipping, spin-coating, flow-coating, etc., using a solution containing the hydrophobic agent.

To manufacture glazing with a hydrophobic coating 30 according to the present invention, it will be possible to use *inter alia*, one of the following three general methods:

(1) the sublayer is deposited on the glass on a glass manufacturing line using the "float" process while the glass is being supported by the bath of molten tin, or in a subsequent step, that is to say on leaving the bath of molten tin, the conversion operations are then carried out, such as bending,

toughening and/or assembling, especially by lamination, in order to obtain plates of glass made up from one or more sheets coated with the sublayer on at least one face, the sublayer or sublayers supported by said plates are then activated and, finally, functionalization by the hydrophobic agent sublayer or sublayers thus activated is carried out. sublayer is generally deposited by PECVD magnetron sputtering;

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- 10 sheets of glass are manufactured by the float (2) process, said glass sheets are then converted operations such as bending, toughening assembling, especially lamination, in order to obtain plates of glass made up from one or more sheets, the 15 sublayer is then deposited on at least one face of the plates thus obtained, and the sublayer or sublayers are then activated, followed by the functionalization by the hydrophobic agent of the sublayer or sublayers thus activated:
- 20 (3) the sublayer is deposited on at least one face of glass sheets obtained upon leaving the float process, these sheets thus coated with the sublayer or sublayers are converted, limiting the techniques used to those that do not damage said sublayer(s) (thereby 25 excluding bending and toughening as conversion operations, but allowing assembling, especially lamination), and the sublayer or sublayers are then activated, followed by the functionalization by the hydrophobic agent of said sublayer or sublayers thus 30 activated.

The present invention also relates to rain-repellent glazing comprising a substrate as defined above or prepared by the process as defined above. Mention may be made of glazing for buildings, including glazing for shower cubicles, glass for electrical household appliances, especially glass-ceramic hobs, glazing for transport vehicles, especially for automobiles and

aircraft, in particular for windshields, side windows, rear windows, wing mirrors, sunroofs, headlamp and rear light optics, and ophthalmic lenses.

5 The following examples illustrate the present invention without however limiting the scope thereof. In these examples, the following abbreviations have been used:

PECVD: plasma enhanced chemical vapor

deposition;

10 SEM: scanning electron microscopy;

AFM: atomic force microscopy; and

AWR: aviation wiping rig.

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EXAMPLE 1: Substrate having a hydrophobic surface according to the invention with a silica sublayer formed by PECVD

(a) Formation and characterization of the hard silica sublayer

A thin silica (SiO_2) layer was deposited on a clean glass (measuring $300 \times 300 \text{ mm}^2$) in a low-pressure PECVD reactor. Before each experiment, the residual vacuum reached in the chamber was at least 5 mPa (5×10^{-5}) 25 mbar). The gas mixture was then introduced into the chamber. The gases used were pure silane (SiH4), nitrous oxide (N_2O) and dilution helium, the respective flow rates of which were 18 sccm, 60 sccm and 60 sccm. The total pressure in the reactor was then set at 9.99 Pa 30 (75 mTorr). In equilibrium, the plasma was struck by biasing the gas diffuser with an average radiofrequency (13.56 MHz) power of 190 W (bias voltage: \sim 45 V). The temperature of the substrate was kept at 25°C. The thickness of silica thus deposited after 270 s 35 about 50 nm.

The surface state of the PECVD silica observed in SEM was characterized by small grains about twenty

nanometers in size, which, in places, formed circular or elongate areas of additional thickness that were hollow at their center.

- 5 The hardness of the silica obtained was characterized using the following two tests:
 - firstly, the layer underwent an abrasion treatment, during which the haze was measured according to the standard ISO 3537; the abrasion was of the Taber type, carried out by means of a CS10F abrasive wheel with an applied force of 4.9 N (500 g). The degree of abrasion was denoted by the number of Taber revolutions. The measured haze values are given in Table 1 below; and
- secondly, the hardness of the silica was assessed by the Airco rating, the value being 10 0.18R in which R is the number of scratches, after a given number of Taber revolutions, in a frame measuring 2.54 cm × 2.54 cm, visible on a photograph with a ×50 magnification. The Airco ratings are also given in Table 1 below.

Table 1: Characterization of the SiO₂ sublayer

State of	Taber revolutions					
abrasion:	50	100	200	300		
Haze	0.55	1.01	1.58	1.75		
Airco	7.48	7.48	6.76	5.86		

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These values characterize a hard SiO2 layer.

(b) Plasma treatment

30 The SiO₂ layer was then subjected to a plasma treatment.

As in the case of the deposition experiments, a residual vacuum of at least 5 mPa (5 \times 10⁻⁵ mbar) was again created in the chamber before the reactive gas

mixture was introduced. The gases used for the surface treatment of the silica were C_2F_6 and oxygen, respective flow rates of which were 120 sccm and 20 sccm. The total pressure in the reactor was then set at 26.66 Pa (200 mTorr). At the equilibrium, the plasma was struck by biasing the gas diffuser with an average radiofrequency (13.56 MHz) power of 200 W voltage: ~ 15 V) for time of 900 s а at room temperature.

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After 15 minutes of C_2F_6/O_2 plasma treatment, the silical layer was highly etched. Its surface had large blisters a few tens of nanometers in size. The microroughness obtained with this highly aggressive plasma (etching) treatment was characterized by AFM, indicating an apparent roughness on the scale of the fluorosilane molecules subsequently grafted onto the silica.

The main microroughness parameters of the PECVD silica 20 measured by AFM are given in Table 2 below.

Table 2

Substrate	ΔZ _{max} * (nm)	R _{rms}	Developed area (2×2) μm^2	Increase (%)
Flat glass	0.5	~ 0.2	4.1	+ 2.5
SiO ₂	10	1.657- 2.116	4.431	+ 10.785
Etched SiO ₂	30	5.981- 7.216	5.5	+ 37

 $^{^*\}Delta Z_{max}$ is the maximum peak/valley amplitude.

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(c) Application of fluorosilane

After the surface of the PECVD silica had been plasma-

treated, a composition was wiped onto the specimens, the composition having been produced 12 hours beforehand in the following manner (the percentages are in weight):

- 5 90% of propanol-2 and 10% of 0.3N HCl were mixed in water; and
 - added to the two aforementioned constituents was 2% of the compound of formula $C_8F_{17}\left(CH_2\right)_2Si\left(OEt\right)_3$ (Et = ethyl).

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The weights per unit area of fluorine grafted onto the surface of the various sublayers, determined by electron microprobe, were:

- on flat glass (with sol-gel

SiO₂ primer sublayer): $0.15 \mu g/cm^2$

- on SiO_2 (PECVD): 0.369 $\mu g/cm^2$

- on etched SiO_2 (PECVD): 1.609 μ g/cm².

The amount of fluorine grafted onto the etched SiO_2 20 sublayer is remarkably high.

- (d) <u>Characterization of the hydrophobic substrate</u> obtained
- 25 The characteristics of the hydrophobic substrate obtained were:
 - droplet contact angle: $\mu_{\text{water}} \ge 105^{\circ}$;
 - optical properties: $T_L = 90.2\%$; $R_L = 8.44\%$; absorption = 1.36%; haze = 0.2%;
- 30 detachment volumes: 13 μl at 90° and 22 μl at 45° (the angles being the angles of inclination of the substrate to the horizontal).
- Next, the above three types of flüorosilane-grafted substrates were subjected to two types of mechanical tests:
 - Taber test using a CS-10F abrasive wheel with a load of 4.9 N (500 g);

- Opel test according to Building Standard EN 1096-2 of January 2001, consisting in applying, to part of the coated surface 9.4 cm in length - this part being called a track - a felt 14 mm in diameter, 10 mm in thickness and $0.52~\rm g/cm^2$ in density, and a load of 39.22 MPa (400 g/cm²), the felt being subjected to a translational movement (50 to-and-fro movements over the entire track length per minute) combined with a rotation of 6 revolutions/minute (1 cycle = 1 to-and-fro movement).

The results of the Opel and Taber tests on the etched and unetched PECVD layers compared with the flat glass are given in Table 3 below.

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Table 3

	MPa (0.4		volutions N (500 g))	
·*.	5000 cycles	100	300	
Control*	95° ± 5°	75° ± 5°	≤ 60°	
PECVD SiO ₂	87° ± 2°	95° ± 1°	83° ± 2°	
Etched SiO2	95° ± 5°	90° ± 1°	74° ± 2°	

*Specimen prepared according to Example 5b of EP 799 873 B1.

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The 87° value in the Opel test (5000 cycles) for the case of the SiO_2 sublayer is not sufficient.

Only the substrate with an etched SiO_2 sublayer results in a good compromise between the Opel test and the Taber test (100 revolutions).

This substrate was therefore tested in the AWR, consisting in moving an aircraft windshield wiper over along 25 cm track in a transverse movement consisting of two to-and-fro movements per

under a load of 0.88 N/cm (90 g/cm) with a water spray of 6 l/h.

A mean angle of about $80^{\circ} \pm 10^{\circ}$ after 1 000 000 cycles was measured, with only 26% of the area not functional $(\mu_{\text{water}} < 60^{\circ})$. The functionality limit was measured to be 1 400 000 cycles, at which the mean angle was about $70^{\circ} \pm 10^{\circ}$ with more than 35% of the area not functional.

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The substrate was also assessed by the following main accelerated environmental tests:

- WOM or Xenon test: 0.55 W/m^2 irradiation at 340 nm;
- 15 QUV: 16 h of UV-B (313 nm) at 70°C + 8 h at 40°C (> 95% residual humidity);
 - NSS: exposure at + 35° C, 50 g/l NaCl at 7 pH according to the IEC 60 068 standard, part 2-11 Ka.
- 20 All the results are given in Table 4.

Table 4

	WOM		QUV		BSN	
	600 h	2000 h	1500 h	3500 h	2 weeks	4 weeks
Control*	105° ± 5°	95° ± 3°	90° ± 5°		65° ±	65° ±
Etched	104° ±	102° ±	105° ±	95° ± 3°	103° ±	103° ±
SiO ₂	5°	3°	5°	33 ± 3	15°	5°

*Specimen prepared according to Example 5b of 25 EP 799 873 B1.

The etched PECVD sublayers made it possible to maintain, in the QUV test, a $\mu_{water} > 80^{\circ} \pm 6^{\circ}$ after 7000 hours of exposure and a $\mu_{water} \ge 96^{\circ} \pm 3^{\circ}$ after 2800 hours of exposure in the WOM.

EXAMPLE 2: Substrate having a hydrophobic surface according to the invention with a silica sublayer deposited by magnetron sputtering

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(a) Formation and characterization of the hard silica layer

This example relates to the grafting of fluorosilane onto an SiO_2 sublayer formed by reduced-pressure magnetron sputtering.

Three types of SiO₂ were produced:

- SiO_2 under a pressure of 200 Pa (2 μ bar); Ar 15 flow rate: 15 sccm; O_2 flow rate: 12 sccm;
 - SiO_2 under a pressure of 400 Pa (4 μ bar); Ar flow rate: 27 sccm; O_2 flow rate: 12 sccm;
 - SiO_2 under a pressure of 800 Pa (8 μ bar); Ar flow rate: 52 sccm; O_2 flow rate: 15 sccm.

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The plasma was ignited by increasing the DC power from 0 to 2000 W at a rate of 20 W/s.

A presputtering operation consisted in applying, for 3 minutes, a 40 kHz pulsed DC power of 2000 W with 4 μs between the pulses.

A target containing 92% silicon and 8% aluminum was sputtered.

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To obtain a 100 nm SiO_2 coating in one pass, the run speed of the substrate beneath the target was: 5.75 cm/min (200 Pa/2 μ bar), 5.73 cm/min (400 Pa/4 μ bar) and 5.53 cm/min (800 Pa/8 μ bar).

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The hardness of the 200 Pa (2 μ bar) and 800 Pa (8 μ bar) magnetron SiO₂ layers was measured as described in the case of the PECVD SiO₂ layers above: measurement of the

haze (in %) during a Taber abrasion test (ISO 3537), Airco rating.

The results are given in Table 5 below.

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Ta:	ble	5

Measured abrasion			Taber rev	olutions	
state:		50	100	200	300
Haze (%)	SiO ₂ -200 Pa	0.55	0.86	1.35	1.48
	SiO ₂ -800 Pa	0.68	0.99	1.48	1.69
70	SiO ₂ -200 Pa	7.84	7.87	7.48	7.3
Airco	SiO ₂ -800 Pa	8.02	7.84	7.12	6.94

These SiO_2 layers produced by magnetron sputtering were 10 hard layers.

(b) Plasma treatment

Magnetron-deposited (400 Pa/4 μ bar and 800 Pa/8 μ bar) silicas were plasma-etched (230 W/300 s) as follows:

- 1) SiO_2 (400 Pa/4 μ bar): 30%-70% SF_6 at 9.99 Pa/75 mTorr;
- 2) SiO₂ (800 Pa/8 μ bar): a) 20% O₂/80% C₂F₆ at 26.66 Pa/200 mTorr; b) 50% O₂/50% C₂F₆ at 20 26.66 Pa/200 mTorr.

(c) Fluorosilane application

The procedure was as described at (c) of Example 1.

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Five specimens were subjected to various tests, as described below:

- I SiO_2 (400 Pa/4 μ bar) sublayer plasma treated according to 1) above and then the fluorosilane wiped on in order to graft it (as described above);
- II SiO $_2$ (400 Pa/4 μ bar) sublayer with no plasma treatment and the fluorosilane wiped on, upon leaving

the magnetron line for preparing the SiO2;

III SiO_2 (800 Pa/8 μ bar) sublayer plasma treated according to 2a) above and then the fluorosilane wiped on;

IV SiO_2 (800 Pa/8 μ bar) sublayer plasma treated according to 2b) above and then the fluorosilane wiped on; and

V SiO₂ (800 Pa/8 μ bar) sublayer with no plasma treatment and the fluorosilane wiped on, upon leaving the magnetron line for preparing the SiO₂.

The results are given in Table 6 below.

Table 6

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Specimen	Etched thickness	detac vol	ter hment ume		μ _{water} ((°)		% haze
	. (mm)	at at 45° 90°	Initial	Opel (5000 cycles)	Taber (1000 rev.)	NSS (3 wk.)	1000 Taber	
I	25	24	14	109.5	101.6	92	108.3	1.09
II				110.4	102.9	92.3		
III	25	24	13	110.3	102.9	95	108.2	
IV	56	23	13	111.1	106.7	90.6	107.1	
V				111	101.4	88.7		

This table shows the very high performance in general, and especially that of test III in the Taber test and test IV in the Opel friction test.

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EXAMPLE 3

The purpose of this example is to compare four hydrophobic glasses:

25 VI specimen prepared according to Example 5b of

EP 799 873 B1;

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VII magnetron-deposited SiO_2 (800 Pa/8 μ bar) sublayer (Example 2) plasma treated with 70 sccm of SF₆, 30 sccm O_2 at 9.99 Pa/75 mTorr, 230 W, 300 s, the fluorosilane being wiped on;

VIII magnetron-deposited SiO₂ (400 Pa/4 μ bar) sublayer (Example 2) plasma treated with 50 sccm of C₂F₆, 50 sccm at 26.66 Pa/200 mTorr, 230 W, 300 s, the fluorosilane being wiped on; and

10 IX fluorosilane application by wiping on, upon leaving the magnetron-deposited (800 Pa/8 μ bar) silica production line.

Various tests were carried out on the specimens thus formed, and the results are given in Table 7 below.

		$\mu_{\mathtt{water}}$	r (°)		%
Specimen	Initial	Taber (100 revs.)	Opel (5000 cycles)	AWR (50 000 cycles)	degraded area (μ _{water} < 60°)
VI	109.6	88.4	104.7	104.8	1.0
VII	111.8	93	103.5	105.0	0.0
VIII	112.5	101.7	104.8	96.0	1.5
IX	112.2	86	108.1	96.4	5.5

Table 7

20 The percentage of degraded area (μ_{water} < 60°) was assessed after 50 000 AWR cycles.

Specimens VI to IX that had undergone 50 000 AWR cycles were subjected to an NSS test in the case of some of them and to a QUV test in the case of the others.

The results are given in Table 8 below.

Table 8

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			μ	water (°)			
NSS						QUV	
Specimen		Number of days				er of h	ours
	0	3	20	50	0	1431	3187
VI	105.8	64.1	25.0		99.0	89.0	87.0
VII	106.9	106.6	105.1	99.4	96.0	99.0	87.0
VIII	99.7	95.7	89.4	84.8	92.0	71.0	61.0
IX	97.8	99.8	90.5	84.1	96.0	79.0	65.0

This shows the remarkable performance of specimen VII in the combined AWR/NSS and AWR/QUV tests.

10 Specimens VIII and IX are slightly inferior to VII in the AWR/NSS test combination and substantially inferior in the AWR/QUV combination, while still being at a high level, unknown before the implementation of the invention.

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EXAMPLE 4

This example describes a particular treatment of the magnetron-deposited (800 Pa/8 μ bar) SiO₂ sublayers.

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This treatment comprised:

- (1) Five minutes, treatment in Ar (80 sccm, 19.98 Pa/150 mTorr), 200 W (35 V bias voltage) in order to reduce any residual roughness;
- 25 (2) Flash surface treatment: duration \leq 60 s (= 60 s in this example), C_2F_6 , SF_6 , O_2 , H_2 ;
 - (3) Fluorosilane application by wiping.

Specimens X to XV are described below by the

characteristics of their treatment step (2):

X: 26.66 Pa/200 mTorr, 230 W, 50 sccm C_2F_6 , 50 sccm O_2 ;

XI: as X, except 100 sccm C_2F_6 ;

XII: as X, except 70 sccm SF_6 and 30 sccm O_2 ;

XIII: 9.99 Pa/75 mTorr, 203 W, 100 sccm SF_6 ;

XIV: 7.99 Pa/60 mTorr, 230 W, 100 sccm O_2 ; and

XV: 13.33 Pa/100 mTorr, 230 W, 75 sccm H_2 .

The amount of grafted fluorine [F] was determined by electron microprobe, and then an Opel friction resistance test was carried out. The results are given in Table 9 below.

15 <u>Table 9</u>

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	Etched	Grafted		μ _{water} (°C)	
Specimen	thickness (nm)	fluorine (µg/cm²)	Initial	Opel (5000 cycles)	Opel (15 000 cycles)
X	10	0.9	109.3	102	93.9
XI	< 5	0.4	104.1	105.5	103.3
XII	16	. 0.8	110.6	103.7	93.6
XIII		0.4	105	104.6	100
XIV	< 5	0.3	106.9	103	101.8
xv	< 5	0.4	111.9	103.9	103.3

These results show that the friction resistance is not directly correlated with the amount of grafted fluorine, or with the roughness of the sublayer (since the etched thicknesses do not exceed 16 nm, the increase in roughness generated by the etching process is in this case negligible). However, the fluorine grafting mode plays a role that depends on the surface treatment.

The invention has been described using the word

"substrate". It should be understood that this substrate may be a bare substrate, but it may also be a substrate already provided with functionalities other than the rain-repellent functionality, in particular thanks to layers, and, in certain cases, the sublayer according to the invention may then already form part of the layers that provide these other functionalities.

CLAIMS

- A substrate, at least one part of the surface of which has been rendered hydrophobic, having for this 5 purpose a hydrophobic surface structure comprising an essentially mineral silicon-containing sublayer and an outer layer of hydrophobic agent grafted onto said sublayer, characterized in that said sublayer received the outer layer of hydrophobic agent although it had a surface that was in an activated state before being brought into contact with said hydrophobic agent.
 - The substrate as claimed in claim 1, characterized in that the sublayer is a hard sublayer.

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- The substrate as claimed in either of claims 1 and 3. 2, characterized in that it is formed by a plate, whether plane or with curved faces, of monolithic or laminated glass, of glass-ceramic or of a thermoplastic, such as polycarbonate.
- The substrate as claimed in claim 3, characterized in that the sublayer of the hydrophobic coating forms part of the substrate, the latter being formed by a plate, whether plane or with curved faces, monolithic or laminated glass or of glass-ceramic, the composition of which, at least on the surface, corresponds to that of the essentially mineral siliconcontaining sublayer.

- The substrate as claimed in claim 4, characterized in that the substrate is a glass dealkalized at least on its surface.
- 35 The substrate as claimed in one of claims 1 to 5, characterized in that said sublayer is formed by a compound chosen from SiO_x , where $x \le 2$, SiOC, SiOCN and Si_3N_4 , it being possible for hydrogen to be

combined in all proportions with SiO_x , where $x \le 2$, SiOC, SiON and SiOCN.

- 7. The substrate as claimed in one of claims 1 to 6, characterized in that the silicon-containing sublayer contains aluminum, in particular up to 8% by weight, or carbon, Ti, Zr, Zn and B.
- 8. The substrate as claimed in one of claims 1 to 7, characterized in that the silicon-containing sublayer when its surface is in the activated state has a thickness of between 20 nm and 250 nm, especially between 30 nm and 100 nm and in particular between 30 nm and 75 nm.

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- 9. The substrate as claimed in one of claims 1 to 8, characterized in that the silicon-containing sublayer has, when its surface is in the activated state, an RMS roughness of between 0.1 nm and 40 nm, in particular between a few nm and 30 nm.
- 10. The substrate as claimed in one of claims 1 to 9, characterized in that the silicon-containing sublayer when its surface is in the activated state has an actual developed area at least 40% greater than the initial plane area.
- 11. The substrate as claimed in one of claims 1 to 10, characterized in that the silicon-containing sublayer 30 when its surface is in the activated state has a hardness such that it does not delaminate after 100 revolutions, and preferably up to 200 revolutions, in the Taber test.
- 35 12. The substrate as claimed in one of claims 1 to 11, characterized in that the outer layer of hydrophobic agent is based on a hydrophobic agent chosen from:
 - (a) alkylsilanes of formula (I):

 $CH_3 (CH_2)_n SiR_m X_{3-m}$ (I)

in which:

- n ranges from 0 to 30, more particularly from 0 to 18;
- m = 0, 1, 2 or 3;
 - R represents an optionally functionalized organic chain; and
- X represents a hydrolyzable residue, such as an OR⁰ residue, where R⁰ represents hydrogen; or a linear, branched or cyclic, especially C₁-C₈, alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue;
 - (b) compounds with grafted silicone chains;
 - (c) fluorosilanes, such as those of formula (II):

 $R^{1}-A-SiR_{p}^{2}X_{3-p} \qquad (II)$

in which:

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- R^1 represents an especially C_1 - C_9 monofluoroalkyl, oligofluoroalkyl or perfluoroalkyl residue; or a monoaryl, oligoaryl or perfluoroaryl residue;
- A represents a hydrocarbon chain, optionally interrupted by a heteroatom such as O or S;
- R^2 represents a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue, or an aryl residue; X represents a hydrolyzable residue, such as an OR^3 residue, where R^3 represents hydrogen or a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue; and
- 30 p = 0, 1 or 2.
 - 13. The substrate as claimed in one of claims 1 to 12, characterized in that the layer of hydrophobic agent has a thickness of between 1 and 100 nm, preferably between 2 and 50 nm.
 - 14. The substrate as claimed in one of claims 1 to 13, characterized in that the layer of hydrophobic agent

has a weight per unit area of grafted fluorine of between 0.1 $\mu g/cm^2$ and 3.5 $\mu g/cm^2$.

- 15. A process for manufacturing a substrate as defined in one of claims 1 to 14, characterized in that a coating layer of hydrophobic agent is deposited, in at least one pass, on the surface of a silicon-containing mineral layer formed at least partly on the surface of the substrate, said deposition of the hydrophobic agent taking place while said surface is in the activated state.
- 16. The process as claimed in claim 15, characterized in that an activated surface of the silicon-containing 15 mineral layer is obtained by depositing it under conditions in which its surface is obtained directly in the activated state.
- 17. The process as claimed in claim 15, characterized in that an activated surface of the silicon-containing mineral layer is obtained by carrying out an activation treatment in at least one pass.
- 18. The process as claimed in one of claims 15 to 17, characterized in that the hydrophobic agent is deposited within the shortest possible time, preferably between 1 second and 15 minutes, after the activated surface has been obtained.
- 19. The process as claimed in either of claims 17 and 18, characterized in that an activation treatment is carried out under conditions that do not go as far as etching, by the use of a plasma or an ionized gas, at reduced or atmospheric pressure, chosen from air, oxygen, nitrogen, argon, hydrogen, ammonia and mixtures

thereof, or by the use of an ion beam.

20. The process as claimed in either of claims 17 and

- 18, characterized in that an activation treatment is carried out under conditions that allow a siliconcontaining layer to be etched, by the use of a plasma of at least one fluorine-containing gas chosen from SF_6 , CF_4 , C_2F_6 and other fluorinated gases, where appropriate combined with oxygen, it being possible for the oxygen to represent up to 50% by volume of the etching plasma.
- 21. The process as claimed in claim 20, characterized in that the activation carried out under conditions that allow the silicon-containing layer to be etched by an activation treatment, which does not cause additional etching but does still modify the chemical nature and/or the electrostatic state of said layer, is monitored.
- 22. The process as claimed in one of claims 15 to 21, characterized in that the silicon-containing layer is deposited, cold, on the substrate by vacuum cathode sputtering, preferably magnetron sputtering and/or ion beam sputtering, or by low-pressure or atmospheric-pressure PECVD (plasma-enhanced chemical vapor deposition), or else deposited hot by pyrolysis.
- 23. The process as claimed in claim 22, characterized in that a layer of SiO₂ is deposited, as siliconcontaining layer, by PECVD, using a mixture of an organic or nonorganic, silicon-containing precursor, such as SiH₄, hexamethyldisiloxane, tetraethoxysilane and tetramethyldisiloxane, and an oxidizer, the subsequent activation being carried out in the same chamber or in a separate chamber.
- 24. The process as claimed in one of claims 15 to 23, characterized in that the fluorosilane layer is deposited by wiping-on, evaporation or spraying of a solution containing the fluorosilane, or by dipping, spin-coating, flow-coating, etc., using a solution

containing the fluorosilane.

- The process as claimed in one of claims 15 to 24 25. for the manufacture of glazing having a hydrophobic coating, characterized in that the sublayer deposited on the glass on a glass manufacturing line using the "float" process while the glass is being supported by the bath of molten tin, or in a subsequent step, that is to say on leaving the bath of molten tin, in that the conversion operations are then carried out, 10 bending, toughening and/or assembling, especially by lamination, in order to obtain plates of glass made up from one or more sheets coated with the sublayer on at least one face, in that the sublayer or 15 sublayers supported by said plates are then activated in that, finally, a functionalization by the hydrophobic agent of the sublayer or sublayers thus activated is carried out.
- 20 The process as claimed in one of claims 15 to 24 for the manufacture of glazing having a hydrophobic coating, characterized in that sheets of glass are manufactured by the float process, in that said glass sheets are then converted by operations such 25 bending, toughening and/or assembling, especially lamination, in order to obtain plates of glass made up from one or more sheets, in that the sublayer is then deposited on at least one face of the plates thus obtained, and in that the sublayer or sublayers are then activated, followed by the functionalization by 30 the hydrophobic agent of the sublayer or sublayers thus activated.
- 27. The process as claimed in one of claims 15 to 24, characterized in that the sublayer is deposited on at least one face of glass sheets obtained upon leaving the float process, in that these sheets thus coated with the sublayer or sublayers are converted, limiting

the techniques used to those that do not damage said sublayer(s), and in that the sublayer or sublayers are then activated, followed by the functionalization by the hydrophobic agent of said sublayer or sublayers thus activated.

28. Rain-repellent glazing comprising a substrate as defined in one of claims 1 to 14 or manufactured by the process as defined in one of claims 15 to 27.

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29. An application of the glazing as defined in claim 28 as glazing for the automotive, aviation, building, electrical household appliance and ophthalmic lens industries.

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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF

MAXIME DURAN, ET AL.

: ATTN: APPLICATION DIVISION

SERIAL NO: NEW U.S. APPLICATION

(BASED ON PCT/FR05/50119)

FILED: HEREWITH

FOR: SUBSTRATE SUCH AS A GLASS

SUBSTRATE WITH A

HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES

PRELIMINARY AMENDMENT

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

Prior to examination of the application please undertake the following changes in the claims.

Amendments to the Claims are reflected in the listing of claims that begins on page 2 of this paper.

Remarks begin on page 10 of this paper.

IN THE CLAIMS

Please amend the claims as follows:

Claim 1 (Currently Amended): A substrate, at least one part of the surface of which has been rendered hydrophobic, having for this purpose a hydrophobic surface structure comprising an essentially mineral silicon-containing sublayer and an outer layer of hydrophobic agent grafted onto said sublayer, characterized in that wherein said sublayer has received the outer layer of hydrophobic agent, said sublayer having although it had a surface that was in an activated state before being brought into contact with said hydrophobic agent.

Claim 2 (Currently Amended): The substrate as claimed in claim 1, eharacterized in that wherein the sublayer is a hard sublayer.

Claim 3 (Currently Amended): The substrate as claimed in either of claims 1 and 2, characterized in that claim 1, wherein it is formed by a plate, whether plane or with curved faces, of monolithic or laminated glass, of glass-ceramic or of a hard thermoplastic, such as polycarbonate.

Claim 4 (Currently Amended): The substrate as claimed in claim 3, characterized in that wherein the sublayer of the hydrophobic coating forms part of the substrate, the latter being formed by a plate, whether plane or with curved faces, of monolithic or laminated glass or of glass-ceramic, the composition of which, at least on the surface, corresponds to that of the essentially mineral silicon-containing sublayer.

Claim 5 (Currently Amended): The substrate as claimed in claim 4, eharacterized in that wherein the substrate is a glass dealkalized at least on its surface.

Claim 6 (Currently Amended): The substrate as claimed in one of claims 1 to 5, eharacterized in that Claim 1, wherein said sublayer is formed by a compound chosen from SiO_x , where $x \le 2$, SiOC, SiON, SiOCN and Si_3N_4 , it being possible for hydrogen to be combined in all proportions with SiO_x , where $x \le 2$, SiOC, SiON and SiOCN.

Claim 7 (Currently Amended): The substrate as claimed in one of claims 1 to 6, characterized in that claim 1, wherein the silicon-containing sublayer contains aluminum, in particular up to 8% by weight, or carbon, Ti, Zr, Zn and B.

Claim 8 (Currently Amended): The substrate as claimed in one of claims 1 to 7, eharacterized in that claim 1, wherein the silicon-containing sublayer when its surface is in the activated state has a thickness of between 20 nm and 250 nm, especially between 30 nm and 100 nm and in particular between 30 nm and 75 nm.

Claim 9 (Currently Amended): The substrate as claimed in one of claims 1 to 8, characterized in that claim 1, wherein the silicon-containing sublayer has, when its surface is in the activated state, an RMS roughness of between 0.1 nm and 40 nm, in particular between a few nm and 30 nm.

Claim 10 (Currently Amended): The substrate as claimed in one of claims 1 to 9, characterized in that claim 1, wherein the silicon-containing sublayer when its surface is in the activated state has an actual developed area at least 40% greater than the initial plane area.

Claim 11 (Currently Amended): The substrate as claimed in one of claims 1 to 10, eharacterized in that claim 1, wherein the silicon-containing sublayer when its surface is in the activated state has a hardness such that it does not delaminate after 100 revolutions, and preferably up to 200 revolutions, in the Taber test.

Claim 12 (Currently Amended): The substrate as claimed in one of claims 1 to 11, characterized in that claim 1, wherein the outer layer of hydrophobic agent is based on a hydrophobic agent chosen from:

(a) alkylsilanes of formula (I):

$$CH_3(CH_2)_nSiR_mX_{3-m}$$
 (I)

in which:

- [[-]] n ranges from 0 to 30, more particularly from 0 to 18;
- [[-]] m = 0, 1, 2 or 3;
- [[-]] R represents an optionally functionalized organic chain; and
- [[-]] X represents a hydrolyzable residue, such as an OR⁰ residue, where R⁰ represents hydrogen; or a linear, branched or cyclic, especially C₁-C₈, alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue;
- (b) compounds with grafted silicone chains;
- (c) fluorosilanes, such as those of formula (II):

$$R^{1}-A-SiR_{p}^{2}X_{3-p}$$
 (II)

in which:

- [[-]] R¹ represents an especially C₁-C₉ monofluoroalkyl, oligofluoroalkyl or perfluoroalkyl residue; or a monoaryl, oligoaryl or perfluoroaryl residue;
- [[-]] A represents a hydrocarbon chain, optionally interrupted by a heteroatom such as O or S;

[[-]] R^2 represents a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue, or an aryl residue; X represents a hydrolyzable residue, such as an OR^3 residue, where R^3 represents hydrogen or a linear, branched or cyclic, especially C_1 - C_8 , alkyl residue; or an aryl residue; or such as a halo, for example chloro, residue; and

[[-]]
$$p = 0$$
, 1 or 2.

Claim 13 (Currently Amended): The substrate as claimed in one of claims 1 to 12, characterized in that claim 1, wherein the layer of hydrophobic agent has a thickness of between 1 and 100 nm, preferably between 2 and 50 nm.

Claim 14 (Currently Amended): The substrate as claimed in one of claims 1 to 13, characterized in that claim 1, wherein the layer of hydrophobic agent has a weight per unit area of grafted fluorine of between 0.1 µg/cm² and 3.5 µg/cm².

Claim 15 (Currently Amended): A process for manufacturing a substrate as defined in one of claims 1 to 14, characterized in that claim 1 comprising depositing a coating layer of hydrophobic agent is deposited, in at least one pass, on the surface of a silicon-containing mineral layer formed at least partly on the surface of the substrate, said deposition of the hydrophobic agent taking place while said surface is in the activated state.

Claim 16 (Currently Amended): The process as claimed in claim 15, characterized in that wherein an activated surface of the silicon-containing mineral layer is obtained by depositing it under conditions in which its surface is obtained directly in the activated state.

Claim 17 (Currently Amended): The process as claimed in claim 15, eharacterized in that wherein an activated surface of the silicon-containing mineral layer is obtained by carrying out an activation treatment in at least one pass.

Claim 18 (Currently Amended): The process as claimed in one of claims 15 to 17, eharacterized in that claim 15, wherein the hydrophobic agent is deposited within the shortest possible time, preferably between 1 second and 15 minutes, after the activated surface has been obtained.

Claim 19 (Currently Amended): The process as claimed in either of claims 17 and 18, characterized in that claim 17, wherein an activation treatment is carried out under conditions that do not go as far as etching, by the use of a plasma or an ionized gas, at reduced or atmospheric pressure, chosen from air, oxygen, nitrogen, argon, hydrogen, ammonia and mixtures thereof, or by the use of an ion beam.

Claim 20 (Currently Amended): The process as claimed in either of claims 17 and 18, characterized in that claim 17, wherein an activation treatment is carried out under conditions that allow a silicon-containing layer to be etched, by the use of a plasma of at least one fluorine-containing gas chosen from SF₆, CF₄, C₂F₆ and other fluorinated gases, where appropriate combined with oxygen, it being possible for the oxygen to represent up to 50% by volume of the etching plasma.

Claim 21 (Currently Amended): The process as claimed in claim 20, characterized in that wherein the activation carried out under conditions that allow the silicon-containing layer

to be etched by an activation treatment, which does not cause additional etching but does still modify the chemical nature and/or the electrostatic state of said layer, is monitored.

Claim 22 (Currently Amended): The process as claimed in one of claims 15 to 21, eharacterized in that claim 15, wherein the silicon-containing layer is deposited, cold, on the substrate by vacuum cathode sputtering, preferably magnetron sputtering and/or ion beam sputtering, or by low-pressure or atmospheric-pressure PECVD (plasma-enhanced chemical vapor deposition), or else deposited hot by pyrolysis.

Claim 23 (Currently Amended): The process as claimed in claim 22, eharacterized in that wherein a layer of SiO₂ is deposited, as silicon-containing layer, by PECVD, using a mixture of an organic or nonorganic, silicon-containing precursor, such as SiH₄, hexamethyldisiloxane, tetraethoxysilane and tetramethyldisiloxane, and an oxidizer, the subsequent activation being carried out in the same chamber or in a separate chamber.

Claim 24 (Currently Amended): The process as claimed in one of claims 15 to 23, characterized in that claim 15, wherein the fluorosilane layer is deposited by wiping-on, evaporation or spraying of a solution containing the fluorosilane, or by dipping, spin-coating, flow-coating, etc., using a solution containing the fluorosilane.

Claim 25 (Currently Amended): The process as claimed in one of claims 15 to 24 claim 15 for the manufacture of glazing having a hydrophobic coating, characterized in that comprising depositing the sublayer is deposited on the glass on a glass manufacturing line using the "float" process while the glass is being supported by the bath of molten tin, or in a subsequent step, that is to say on leaving the bath of molten tin, in that the conversion

operations are then carried out, such as bending, toughening and/or assembling, especially by lamination, in order to obtain plates of glass made up from one or more sheets coated with the sublayer on at least one face, in that the sublayer or sublayers supported by said plates are then activated and in that, finally, a functionalization by the hydrophobic agent of the sublayer or sublayers thus activated is carried out.

Claim 26 (Currently Amended): The process as claimed in one of claims 15 to 24 claim 15 for the manufacture of glazing having a hydrophobic coating, characterized in that wherein sheets of glass are manufactured by the float process, in that said glass sheets are then converted by operations such as bending, toughening and/or assembling, especially lamination, in order to obtain plates of glass made up from one or more sheets, in that the sublayer is then deposited on at least one face of the plates thus obtained, and in that the sublayer or sublayers are then activated, followed by the functionalization by the hydrophobic agent of the sublayer or sublayers thus activated.

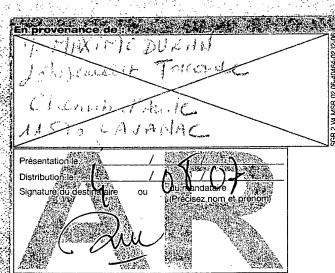
Claim 27 (Currently Amended): The process as claimed in one of claims 15 to 24, characterized in that claim 15, wherein the sublayer is deposited on at least one face of glass sheets obtained upon leaving the float process, in that these sheets thus coated with the sublayer or sublayers are converted, limiting the techniques used to those that do not damage said sublayer(s), and in that the sublayer or sublayers are then activated, followed by the functionalization by the hydrophobic agent of said sublayer or sublayers thus activated.

Claim 28 (Currently Amended): Rain-repellent A rain-repellent glazing comprising a substrate as defined in one of claims 1 to 14 or manufactured by the process as defined in one of claims 15 to 27 claim 1.

Claim 29 (Currently Amended): An application of the glazing as defined in claim 28 as A glazing for the automotive, aviation, building, electrical household appliance and ophthalmic lens industries comprising the rain-repellent glazing of claim 28.

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Signed this 19th day of September 2007

C. E. SITCH

Managing Director - UK Translation Division For and on behalf of RWS Group Ltd

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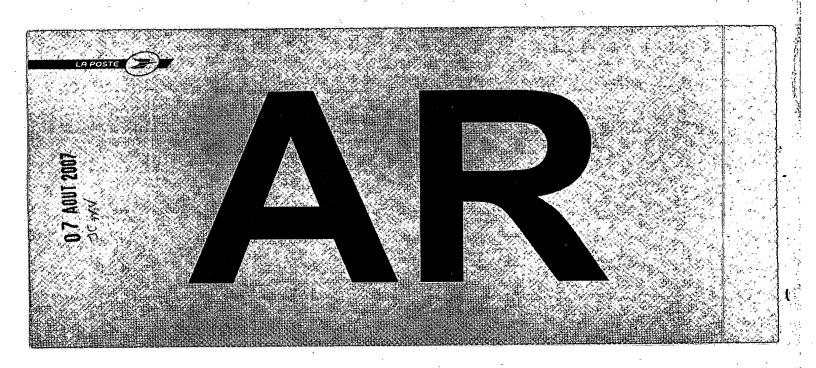
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Declaration and Power of Attorney for Patent Application Déclaration et Pouvoirs pour Demande de Brevet

French Language Declaration

En tant l'inventeur nommé ci-après, je déclare par le présent acte que:	As a below named inventor, I hereby declare that:
Mon domicile, mon adresse postale et ma nationalité sont ceux figurant ci-dessous à côté de mon nom.	My residence, mailing address and citizenship are as stated next to my name.
Je crois être le premier inventeur original et unique (si un seul nom est mentionné ci-dessous), ou l'un des premiers co-inventeurs originaux (si plusieurs noms sont mentionnés ci-dessous) de l'objet revendiqué, pour lequel une demande de brevet a été déposée concernant l'invention intitulée	I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor(if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.
SUBSTRAT, NOTAMMENT VERRIER, A SURFACE HYDROPHOBE, AVEC UNE DURABILITÉ AMELIORÉE DES PROPRIÉTÉS HYDROPHOBES	SUBSTRATE, SUCH AS A GLASS SUBSTRATE, WITH A HYDROPHOBIC SURFACE AND IMPROVED DURABILITY OF HYDROPHOBIC PROPERTIES
et dont la description est fournie ci-joint à moins	the specification of which
☐ ci-joint	is attached hereto.
a été déposée le 23/02//2005 sous le numéro de demande des États-Unis ou le numéro de demande international PCT/FR2005/050119 et modifiée le (le cas échéant).	was filed on 23/02//2005 as United States Application Number or PCT International Application Number PCT/FR2005/050119 and was amended on (if applicable)
Je déclare par le présent acte avoir passé en revue et compris le contenu de la description ci-dessus, revendications comprises, telles que modifiées par toute modification dont il aura été fait référence ci-dessus.	I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.
Je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations.	I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119(a)-(d) ou § 365(b) du Code des États-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur ou, en vertu du Titre 35, § 365(a) du même Code, sur toute demande internationale PCT désignant au moins un pays autre que les États-Unis et figurant ci-dessous et, en cochant la case, j'ai aussi indiqué ci-dessous toute demande étrangère de brevet, tout certificat d'inventeur ou toute demande internationale PCT ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

Prior Foreign Application(s)
Demande(s) de brevet antérieure(s) dans un autre pays.

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Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 119(e) du Code des États-Unis, de toute demande de brevet provisoire effectuée aux États-Unis et figurant ci- dessous.

(Application No.) (Filing Date)
(N° de demande) (Date de dépôt)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des États-Unis, de toute demande de brevet effectuée aux États-Unis, ou en vertu du Titre 35, § 365(c) du même Code, de toute demande internationale PCT désignant les États-Unis et figurant ci-dessous et, dans la mesure où l'objet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande antérieure américaine ou internationale PCT, en vertu des dispositions du premier paragraphe du Titre 35, § 112 du Code des États-Unis, je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la demande antérieure et la date de dépôt de la demande nationale ou internationale PCT de la présente demande:

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(N° de demande) (Date de dépôt)

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Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d'une incarcération, ou des deux, en vertu de la § 1001 du Titre 18 du Code des États-Unis, et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby claim foreign priority under Title 35, United States Code, § 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed

	Priority Cla	imed
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(Day/Month/Year Filed)	Yes	No
(Jour/Mois/Année de dépôt)	Oui	Nor
I hereby claim the benefit under Titl	e 35, United S	States
Code, §119(e) of any United States	provisional	
application(s) listed below.		

(Application No.) (Filing Date) (N° de demande) (Date de dépôt)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application,

(Status: Patented, Pending, Abandoned) (Statut : breveté, en cours d'examen, abandonné)

(Status: Patented, Pending, Abandoned) (Statut : breveté, en cours d'examen, abandonné)

I hereby declare that ail statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

French Language Declaration

POUVOIRS: En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) suivant(s) pour qu'ils poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire s'y rapportant avec l'Office des brevets et des marques: (mentionner le nom et le numéro d'enregistrement).

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact ail business in the Patent and Trademark Office connected therewith: (list name and registration number)



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(Fournir les mêmes renseignements et la signature du septième co-inventeur et de tout co-inventeur supplémentaire.)

(Supply similar information and signature for seventh and subsequent joint inventors.)